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Nationwide Urban Runoff Program
Priority Pollutant Analysis
Final Report

Metropolitan Washington Council of Governments
DC

Prepared for

Environmental Protection Agency, Washington, DC

30 Sep 82

U.S. Department of Commerce
National Technical Information Service

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FINAL REPORT
PRIORITY POLLUTANT ANALYSIS
CONTRACT # P003208-01-03

PREPARED FOR:

METROPOLITAN WASHINGTON COUNCIL OF GOVERNMENTS
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This report was developed based on the analytical analyses performed to establish the presense and concentration of priority pollutants. It includes quality assurance/control documentation, analytical results and conclusion based on findings.

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JTC Environmental Consultants, Inc.

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October 6, 1982

Mr. Cameron Wiegand
Metropolitan Washington
Council of Governments
1875 Eye Street, N.W.
Suite 200
Washington, D. C. 20006

Re: Final Report, Contract #POO 3208-01,
Priority Pollutant Analysis

Dear Mr. Wiegand:

JTC Environmental Consultants, Inc., is pleased to submit its final data report and summary prepared for the Metropolitan Washington Council of Governments. In addition to the analytical results, we have provided examples of our quality control and quality assurance procedures, including Chain of Custody records, sample receipt forms, and laboratory performance evaluation results to assist you in your data review.

It has been our pleasure to work with COG on this project, and we hope to have the opportunity to serve you again.

Sincerely,

John T. Cookson

John T. Cookson, Jr., Ph.D.
President

JTC:jr
encls.

JTC Environmental Consultants, Inc.

7979 OLD GEORGETOWN ROAD · BETHESDA, MARYLAND 20814 · (301) 656-5850

November 11, 1982

Metropolitan Washington
Council of Governments
ATTN: Mr. Cameron Wiegand
1875 Eye Street, N.W., Suite 200
Washington, D.C. 20006

Re: Contract No. P00 3208-01-03
Priority Pollutant Analysis

MEMORANDUM

Dear Mr. Wiegand:

Further review of the final report for the above-named contract has disclosed three typographic errors. Would you be kind enough to make the corrections noted below:

Sample	Parameter	Correct reading
UR 10	Zn	136
UR 06	VOA blank	as noted
UR 09	γ-BHC (not δ-BHC)	trace

If you have any questions, please call us at the number noted above.

Sincerely yours,

JTC ENVIRONMENTAL
CONSULTANTS, INC.

cc: Mr. Clifford Mitchell
D,D,D & N

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INTRODUCTION

The analytical results presented here were performed under the direction of the Metropolitan Washington Council of Governments (COG). The general purpose of this program is to provide information on water quality effects of different land use activities. It is only though an understanding of pollutant discharge relationships with various land use activities that control measures and the application of best management practices can be cost effective. The analytical analyses presented here were performed to establish the presence and concentration of the 129 priority pollutants designed in 1976 by the U.S. EPA with the exception of asbestos.

This section of the report designates the general areas of responsibility and provides a brief overview on the analytical procedures. The second section of this report provides a summary on the analytical results with a brief conclusion regarding the findings. Section three sets forth the quality assurance and summarizes the surrogate spike and matrix spike results. Finally, the appendix provides detailed records on chain of custody, sample log, instrumentation records, and raw data sheets.

At the request of COG representatives, JTC Environmental Consultants, Inc., (JTC) prepared appropriate containers for the specified sampling episode. These containers were picked up by the COG field team, and after sample collection delivered to JTC with appropriate chain of custody as presented in Appendix A. Chain of custody records show the point of origin of the sample and documents its transfer to JTC. At that time the samples are logged onto the JTC laboratory receipt form and assigned an internal JTC log number. This record also indicates the condition of the sample on arrival. The sample receipt form is illustrated in Section B of Appendix A. Samples were placed in appropriate storage facilities at controlled temperatures or extracted and concentrated for appropriate analyses.

Samples designed for organic analyses were extracted with organic solvents and subjected to analysis by gas chromatography for the pesticide fraction and GC/MS for the base neutral and acid fractions. Laboratory blanks were extracted and analyzed under identical conditions. Samples designated for volatile analyses were analyzed by the purge and trap method coupled with GC/MS.

Metals were analyzed by atomic absorption spectrophotometry. The analytical protocols are detailed in EPA method No. 624, and 625 for the organic priority pollutants and EPA procedures as set forth in Report Number 600/4-79-020 for the metals, cyanide and phenol. A summary of the methodology is presented in Table 1.

TABLE 1

TABLE OF METHOD REFERENCES

ANALYTE(S)	INSTRUMENT	METHOD NO.	REFERENCE
Volatile Organics	GC/MS with purge & trap	EPA 624	Combined Federal Register V.44, no.233 Dec 3, 1979
Base/Neutral & Acid Extractables	GC/MS via direct injection	EPA 625	
Antimony	AA, furnace	EPA 204.1	EPA-600/4-79-020, Methods for Chemical Analysis of Water and Wastes, March, 1979
Arsenic	AA, furnace	EPA 206.2	
Beryllium	AA, furnace	EPA 210.2	
Cadmium	AA, furnace	EPA 213.1	
Chromium	AA, furnace	EPA 218.2	" "
Lead	AA, furnace	EPA 239.2	" "
Mercury	AA, cold vapor manual	EPA 245.1	" "
Nickel	AA, furnace	EPA 249.2	" "
Selenium	AA, furnace	EPA 270.2	" "
Silver	AA, furnace	EPA 272.2	" "
Thallium	AA, furnace	EPA 279.2	" "
Zinc	AA, flame	EPA 289.1	" "
Cyanide	Colorimetric	EPA 335.3	" "
Phenols	Spectrophotometric	EPA 420.3	" "
EP Toxicity	Extraction	SW 846	Combined Federal Register V.45, no.98 p. 33127

ANALYTICAL RESULTS

The following presents a summary with more details provided in the raw data sheets in Appendix B. The organic analyses are summarized according to sample numerical order of the UR series followed by alphabetical order for named samples (Tables 2-8). Immediately following the analyses summary is a listing of field blanks using the same numerical order sequence.

In general, the samples submitted with the UR series designation contained a low level of pesticides. Although pesticides are found to be present in almost all samples, they are at levels less than what is normally quantitated. For this report, concentrations are reported less than 0.1 µg.1, No pesticides were detected in any of the field blanks.

Only two samples contained detectable levels of priority pollutants in the acid fraction. These are samples UR-13 and UR-17. Each contained 4-nitrophenol and phenol. However, these levels are below the normally reported detection limit and the compounds are also present at low concentrations in the field and laboratory blanks. Thus, samples UR-13 and UR-17 contained no acid fraction priority pollutants after correcting for blanks.

The only significant pollutant category to occur in the base neutral fraction is phthalates. Phthalate concentrations ranged from just detectable to 150 µg/l for sample UR-3. The field blank did not contain any appreciable amount of phthalates and laboratory blanks illustrated only typical background contamination levels. When correcting the phthalate concentrations found in samples for the laboratory blanks, no significant levels would be reported except for possibly sample UR-3.

Except for methylene chloride no significant level of volatiles were found. The interpretation of data for the low level concentrations is difficult because of low level contamination in the volatile field blanks. It appears that the water source utilized for field blanks by the collection team contained methylene chloride and other volatile organics at levels less than 10 µg/l. None of these contaminants were present in any of JTC's laboratory blanks except for trace amounts of methylene chloride. Low level methylene chloride is typically found in laboratory blanks. This data supports the conclusion that water utilized for the field blanks (which was from a different source than that utilized by the JTC laboratory) contained trace amounts of volatile priority pollutants. Since the volatile organic

content of samples were usually at trace amounts, it appears that much of that reported could be a result of field contamination.

In addition to the EPA listed priority pollutants, several organic compounds were found and tentatively identified in Table 9. These compounds could not be quantitated with regard to concentration since standards were not available for quantitation. Their identification and reported presence is made possible through our in-house data bank and computer search facilities.

Measurable levels of phenols were found in samples labeled Burke Pond, Wesleigh Sludge and several of the UR sample series. The highest concentration level reported was 22 µg/l. Cyanide was usually below detection limit but was found at a level of 12 µg/l in sample UR-10 and UR-15.

Table 10 summarizes the inorganic water quality parameters, along with the maximum contaminant levels (MCL) set by the Interim Primary Drinking Water Standards listed where applicable. Samples containing concentrations of specific metals which equalled or exceeded MCL's are: UR 5 (Se), UR 6 (Se), UR 7 (As, Cu, Se), UR 9 (Se), UR 10 (Se), UR 13 (Se), UR 15 (Cu, Se), and UR 19 (Se). Certain metals which were analyzed (Be, Ni, Sb, Tl, and Zn) have no MCL set, but all of these were found in one or more of the samples.

TABLE 2
SAMPLES
SUMMARY OF ORGANIC PRIORITY POLLUTANT COMPOUNDS

SAMPLE NAME NO.	VOLATILE µg/l	BASE NEUTRAL µg/l	ACID µg/l	PESTICIDES µg/l
UR-3	Methylene chloride 46 Methylchloride <10	bis (2-ethylhexyl phthalate 150 di-n-butylphthalate < 10	ND	aldrin <0.1 α - BHC <0.1 γ - BHC <0.1 δ - BHC <0.1
UR-5	1,1,1-Trichlorethane <10 1,1-dichloroethylene <10	bis (2-ethylhexyl phthalate < 10 di-n-butylphthalate < 10	ND	Aldrin <0.1 x-endo-sulfan <0.1 heptachlor <0.1 heptachlor epoxide <0.1 α - BHC <0.1 β - BHC <0.1 δ - BHC <0.1
UR-6	1,1,1-trichloroethane <10 Chloro form <10 1,1 dichloroethylene <10 Methylene chloride <10 ↑ BLANK FOR VOA	isophorone < 10 bis (2-ethylhexyl) phthalate < 10 di-n-butylphthalate < 10 chrysene < 10	ND	α - BHC <0.1

ND = Not detected; NA = Not analyzed

TABLE 3
SAMPLES
SUMMARY OF ORGANIC PRIORITY POLLUTANT COMPOUNDS

SAMPLE NAME NO.	VOLATILE µg/l	BASE NEUTRAL µg/l	ACID µg/l	PESTICIDES µg/l
UR-7	Chlorobenzene <10 Methylene chloride 12	bis(2-ethylhexyl) phthalate <10 di-n-butylphthalate <10	ND	aldrin <0.1 α - endo-sulfan <0.1 heptachlor <0.1 α - BHC <0.1 β - BHC <0.1 γ - BHC <0.1 δ - BHC <0.1
UR-9	1,1 dichloroethylene <10 methylene chloride 43 methylchloride <10	isophorone <10 bis(2-ethylhexyl phthalate <10	ND	α - BHC <0.1 β - BHC ~~<0.1~~ Trace
UR-10	1,1 dichloroethylene <10 Methylene chloride 39 Methylchloride <10	isophorone <10 bis(2-ethylhexyl) phthalate <10	ND	heptachlor <0.1 α - BHC <0.1 β - BHC <0.1 γ - BHC <0.1 δ - BHC <0.1
UR-13	Methylene chloride 20	ND	2,4-dimethyl phenol <10 phenol <10	aldrin <0.1 4,4-DDT <0.1 α - endo-sulfan <0.1 α - BHC <0.1 β - BHC 0.1 γ - BHC 0.1

TABLE 4
SAMPLES

SUMMARY OF ORGANIC PRIORITY POLLUTANT COMPOUNDS

SAMPLE NAME NO.	VOLATILE µg/l	BASE NEUTRAL µg/l	ACID µg/l	PESTICIDES µg/l
UR-15	1,1,1 trichloroethane <10 methylene chloride 12 methyl chloride <10	bis(2-ethylhexyl) phthalate <10 di-n-butylphthate <10 diethylphthalate <10	ND	α - endo-sulfan 0.1 heptachlor 0.1 α - BHC 0.1 γ - BHC 0.1 δ - BHC 0.1
UR-17	methylene chloride 127 methylchloride <10	bis(2-ethylhexyl) phthalate 47 di-n-octylphthalate <10	4-nitro-phenol <10 phenol <10	aldrin <0.1 α - BHC <0.1 γ - BHC <0.1 δ - BHC <0.1
UR-19	methylene chloride 14	bis (2-ethylhexyl) phthalate <10 di-n-octylphthalate <10	ND	ND

TABLE 5

SAMPLES

SUMMARY OF ORGANIC PRIORITY POLLUTANT COMPOUNDS

	VOLATILE µg/l	BASE NEUTRAL µg/l	ACID µg/l	PESTICIDES µg/l
urke Pond Sludge	NA	ND	ND	ND
urke Pond Volatiles	ND	NA	NA	NA
akeridge	ND	ND	ND	α -BHC < 0.1 γ -BHC < 0.1
ttle Seneca	ND	bis(2-ethylhexyl phthalate) 19	ND	ND
ockville Pond latiles	methylene chloride 19	NA	NA	NA
ckville Pond Sludge	ND	ND	ND	α-BHC < 0.1 β-BHC < 0.1 γ -BHC < 0.1
esleigh Sludge	NA	ND	ND	heptachlor < 0.1 γ - BHC 0.2 γ - BHC < 0.1
sleigh Water	ND	ND	ND	heptachlor <0.1

TABLE 6
Field Blanks

SUMMARY OF ORGANIC PRIORITY POLLUTANT COMPOUNDS

ld Blanks	VOLATILE µg/l	BASE NEUTRAL µg/l	ACID µg/l	PESTICIDES µg/l
3	methylene chloride 178 methyl chloride 18	NA	NA	NA
5	1,1,1-trichloroethane <10 1,1 - dichlotoethylene 11 methylene chloride 121	NA	NA	NA
7	1,1-trichloroethane <10 chloroform <10 ethybenzene <10 methylene chloride 39 toluene 15	NA	NA	NA
10	methylene chloride 290 methyl chloride <10	NA	NA	NA
13	ND	NA	NA	NA
15	1,1,1-trichloroethane <10 chloroform <10 methylene chloride <10 tetrachloroethylene <10 toluene 14	NA	NA	NA

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TABLE 7
FIELD BLANKS
SUMMARY OF ORGANIC PRIORITY POLLUTANT COMPOUNDS

	VOLATILE µg/l	BASE NEUTRAL µg/l	ACID µg/l	PESTICIDES µg/l
R-17 (Extractables	ND	bis(2-ethylhexyl) phthalate 66 di-n-butyl phthalate <10	4-nitrophenol 10 phenol 10	ND
R-19	1,1-dichloroethylene 10 methylene chloride 260 methyl chloride 10	NA	NA	NA
arke Pond	Methylene chloride 147	NA	NA	NA
ockville Pond	Methylene chloride 828	NA	NA	NA

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TABLE 8

SUMMARY OF ORGANIC PRIORITY POLLUTANT COMPOUNDS

	VOLATILE µg/l	BASE NEUTRAL µg/l	ACID µg/l	PESTICIDES µg/l
blank 8/10/81	NA	bis(2-ethylhexyl) phthalate 86	NA	NA
blank 10/14/82	methylene chloride 18	bis(2-ethylhexyl) phthalate <10	4-nitrophenol <10	NA
blank 8	NA	bis(2-ethylhexyl) phthalate <10	NA	NA
blank 8	methylene chloride 10	bis(2-ethyhexyl) phthalate 21 di-n-buthylphthalate 2 di-n-octylphthalate <1	ND	ND
blan 8	methylene chloride 18	bis(2-ethylhexyl) phthalate <10	ND	ND

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TABLE 9

Non-Priority Pollutant Compounds Tentatively Identified

(No quantitation is listed since these are not normally part of the analysis)

SAMPLE	COMPOUND NAME	FRACTION
UR-3	Chloroacetic acid	volatile
UR-10	N,N,N^1,N^1 - Tetramethyl-methanediamine	base/neutral
	3-Pentanamine	base/neutral
UR-13	1,2-Propanediamine	volatile
UR-15	3,4-Dimethyl-3-hexene-2-one	base/neutral
UR-17	2-Propanone	volatile
Burke Pond	N-Butylbenzenesulfonamide	base/neutral
Lake Ridge	Butanoic acid, 1-methylpropyl ester	acid and base/neutral
Little Seneca	Butanoic acid, 1-methylpropyl ester	base/neutral
	Nonanedioic acid, bis (2-ethylhexyl) ester	base/neutral
Rockville Pond	2-Butyltetrahydrofuran	base/neutral
Sludge	N-Butylbenzenesulfonamide	base/neutral

TABLE 10

SUMMARY OF INORGANIC WATER QUALITY PARAMETERS[1] (All units are in ppb or ug/l

	Ag µg/l	As µg/l	Be µg/l	Cd µg/l	Cr µg/l	Cu µg/l	Hg µg/l	Ni µg/l	Pb µg/l	Sb µg/l	Se µg/l	Tl µg/l	Zn µg/l	CN µg/l	Phenols µg/l
-UR 3	ND[1]	1	ND	1	10	6	ND	65	18	ND	ND	ND	42	ND	12
-UR 5	ND	ND	35	ND	ND	2	0.6	44	18	ND	68	1	ND	ND	ND
-UR 6	ND	ND	20	ND	ND	2	0.6	119	17	9	40	8	25	6	16
-UR 7	ND	50	1	3	6	50	0.6	8	7	3	17	ND	600	6	ND
-UR 9	ND	ND	44	ND	2	5	ND	108	20	7	30	8	21	6	10
-UR 10	ND	ND	35	1	3	7	ND	50	20	8	59	14	136 ~~36~~	12	22
-UR 13	ND	3	49	3	16	46	0.6	37	26	ND	24	5	122	ND	16
-UR 15	ND	43	1	2	20	60	0.8	16	38	3	17	ND	ND	12	13
-UR 17	ND	2	ND	1	10	13	ND	1	18	ND	ND	ND	42	ND	10
-UR 19	ND	ND	34	ND	ND	1	ND	65	18	ND	77	5	ND	ND	ND
-Burke Pond Sludge	ND	ND	ND	ND	ND	ND	ND	8	1	ND	ND	ND	ND	ND	19
-Lakeridge	ND	ND	ND	ND	3	1	ND	5	8	ND	ND	ND	26	ND	ND
-Little Seneca	ND	ND	ND	ND	2	3	ND	ND	2	ND	ND	ND	34	ND	ND
-Wesleigh Water	ND	ND	ND	ND	2	ND	ND	ND	ND	ND	ND	ND	15	ND	ND
-Wesleigh Sludge	ND	ND	ND	ND	ND	ND	ND	32	1	ND	ND	ND	300	ND	20
Typical Detection Limits	0.5	0.5	0.2	0.1	1.0	1.0	0.2	1.0	1.0	3.0	2.0	1.0	5.0	5.0	5.0
IPW[2] Standards	50	50	NA[3]	10	50	50	2	NA	50	NA	10	NA	NA	NA	NA

1. ND = Not Detected
2. EPA Interim Primary Drinking Water Standards
3. NA = Not Applicable

QUALITY ASSURANCE

There are several basic divisions to the Quality Assurance Program applied to this study. First is the overall base-line laboratory assurance which outlines aspects of our program related to equipment, reagents, standards, glassware, and personnel training. Much of this is detailed in JTC's Quality Assurance Program Plan in Appendix C. The use of standard operating protocols which are followed by all personnel is a part of this base line assurance. In addition, it details the steps to assure that personnel are specifically trained on particular analytical methods prior to their being given responsibility. Equipment and major items or apparatus in the JTC laboratory are grouped according to analytical use. Each group has a major responsible person and one or more "use logs" that are currently maintained and contain such information as: operator, analysis/mode, project, date, duration, and comments. This latter category includes descriptions of problems, solutions and/or maintenance of the equipment. In addition, a file is kept of all manufacturers information for quick access when needed.

The second stage of our Quality Assurance Program deals with sample handling. Since samples were collected by personnel outside of JTC's responsibility, our sample handling assurance dealt with sample receiving and internal tracking within the laboratory. The samples are received by JTC with a sample chain of custody record. After formal receipt of the sample, it is tagged and logged into JTC's records. Any further handling of the sample or changes in its conditions are also entered into appropriate log bookds. For example, removal of a sample for extraction or concentration is noted with the analyst's name. When the sample is either terminated or discarded, it is so logged and initialed.

The third phase of our Quality Assurance Program is the analytical method assurance. Prior to the analytical determination, standards were made, equipment assembled and checked, and methodology applied. A special note here is the preperformance evaluation of the GC/MS. Prior to any analytical work the GC/MS is first carefully calibrated utilizing perfluorotributylamine (FC43). Performance of the instrument is checked and verified through the ability to chromatograph and analyze decafluorotriphenylphosphine according to the mass spectra requirements of the EPA procedures. The calibration data and standards are permanently documented on magnetic tape and on hard copy. Other equipment such as gas chromatographs and atomic absorption units are standardized and strip charts maintained.

After method preparation, the method is tested to ensure proper preparation and capability to recover organic parameters according to EPA protocols. To evaluate this capability, performance Evaluation samples submitted by NURP were analyzed according to their insturctions for volatile organics, pesticides, trace metals, cyanide, and phenol. This performance evaluation was submitted to John A Winter, Chief Quality Assurance Branch, Environmental Monitoring and Support Laboratory, USEPA, Cincinnati. Details on the NURP Performance Evaluation and data report are contained in the Appendix D.

The final aspect of our analytical quality assurance is continuing method challenging. For all samples submitted, surrogate spikes were added for the purpose of monitoring the efficiency of each specific analytical run. The term surrogate spike is used for any of several compounds that have been deuterated or fluorinated which are not normally found in nature. These surrogate spikes were added to samples just prior to the analytical procedure. The surrogate spike recovery varies depending upon the specific spike, analytical fraction being evaluated, and the nature of the sample. Recoveries for the volatile surrogate spike D-6 benzene and D-8 toluene generally run from 80-100 percent while those of D-6 phenol and 2-fluorophenol in the acid fraction are usually in the 40 percent range. For most cases, typical surrogate spike recoveries were found for these samples except for an occasional low recovery with the acid fractions.

Another part of the continuing method challenging is through the use of matrix spikes. Matrix spike analyses are performed by splitting a sample and analyzing the first portion of this sample as received by the laboratory. To the second portion, a spike of the priority pollutants is added. Any matrix interferences would be indicated by the percent recovery of the spike sample after correcting for the presence of any priority pollutants originally present in the sample. As part of our Quality Control program, matrix spike analyses were prepared at a frequency of one (1) per 20 samples analyzed for organic priority pollutants. The quality control report for matrix spike analyses is illustrated in Appendix D. Percent recoveries were usually 70-80 percent with an overall range of 31-142 percent recovery.

The last aspect of challenging is the analyses of duplicate samples. At periodic intervals (one (1) per 20 samples) a sample is split within our laboratory and analyzed as a duplicate. From this duplicate analysis, the relative percent difference is established. These quality control reports are illustrated in Appendix D showing a good reproducibility with relative percent differences of 0-34 percent.